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Chemical Characterization of an Envelope C Sample from Hanford Tank 241-AN-102

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SUMMARY

An approximately 14.25 L sample from Hanford waste tank 241-AN-102 was received at the Savannah River Technology Center (SRTC) and chemically characterized. Prior to characterization the sample was diluted to ~6 M sodium concentration. The filtered supernatant liquid, the total dried solids of the diluted sample, and the washed insoluble solids obtained from filtration of the diluted sample were analyzed.

A mass balance calculation of the three fractions of the sample analyzed indicate the analytical results appear relatively self-consistent for major components of the sample. However, some inconsistency was observed between results were more than one method of determination was employed and for species present in low concentrations. An analysis of the organic complexants appears to be consistent with the TOC result. Some evidence was found to indicate the possible contamination of the first shipment of 241-AN-102 samples received at SRTC with Cm^{244} and possibly Am^{241} and plutonium isotopes. The comparison to previous analyses of samples from 241-AN-102 indicates general agreement with the current analytical results. The comparison of the solids analysis showed large deviations attributed to differences in obtaining the solids from the bulk sample.

INTRODUCTION

The BNFL River Protection Project contracted SRTC to provide pretreatment development and testing services to support the BNFL mission to treat Hanford tank waste. As part of the program, SRTC received radioactive Hanford tank waste samples to allow testing of the pretreatment processes with actual waste samples. The first step in this program entails detailed characterization of the radioactive waste samples. The characterization data provides a basis for rational development of pretreatment processes, determination of reagent requirements, and development of physical design parameters for the pretreatment plant.

The characterization portion of the STRC program was conducted under an approved task and quality assurance plan.[1,2,3] The results and the associated uncertainties presented provide a description of the sample received at SRTC. The highly radioactive nature of the samples adds complexity to the analysis. Sub-sampling, large dilutions, and remote handling potentially add error to the analytical accuracy. Replicate sample analysis and submission of standards allow some definition of the magnitude of this error. However, the error associated with obtaining small samples from large non-homogenized waste tanks will be significant. Recent experience at SRS indicates a combined sampling and analytical error on the order of 15 - 20% associated with obtaining small samples from a <u>well</u> <u>mixed</u> waste tank.[4]

The data presented in this report documents the chemical characterization of a ~14.25 L sample of Hanford waste tank 241-AN-102.

EXPERIMENTAL

Sample History

A total of 32 samples of 241-AN-102 were received at SRTC in four shipments between October 1998 and February 1999. Table 1 lists the sample jar labels from the 32 samples received at SRTC.[5] Table 2 shows the source for each jar taken from the shipping information. Initially, the 8 jars received in each shipment were composited into a 4 liter bottle.

The four composite samples formed from the 4 shipments each contained a very small amount of fine, light colored solids. Several of the jars in the third shipment contained a significant quantity (up to 30 volume percent) of crystalline solids in addition to a small amount of the light colored solids. The total volume of sample from 241-AN-102 received at SRTC was ~14.25 L.

A sample designated as "Small C" was obtained from the composite of the first shipment. Approximately 1.5 L of the first shipment composite was diluted with ~750 mL of 0.01 M NaOH producing ~2.25 L of a ~6 M sodium solution. The diluted Small C sample formed was then dead-end filtered through a 0.45 μ disposable nylon filter. The density and weight percent solids were measured on the diluted Small C sample and the filtrate from the diluted Small C sample. Samples of the total dried solids of the diluted Small C sample, the filtered supernatant liquid from the diluted Small C sample, and dried insoluble solids collected during filtration of the diluted Small C sample were prepared for analysis. The insoluble solids were washed on the filter with two 15 mL portions of 0.01 M NaOH to displace interstitial supernatant liquid. The Small C sample was fully characterized. The filtrate from the Small C sample was used for Sr/TRU precipitation beaker tests.

A full composite of the 241-AN-102 material was made by combining the remaining material of the first shipment composite and all of the other 3 shipments composites in a 25 L carboy. The full composite was designated as "Large C". The carboy was equipped with air-powered mixer. To ensure thorough mixing of the Large C composite, the mixer speed was adjusted to provide a vortex at the top of the carboy. While still mixing, the Large C composite was pumped into 4 smaller containers. The weight percent solids were measured on each of the 4 containers of Large C in preparation for dilution. The sodium concentration and other select analyses were measured on samples of the filtered supernatant of Large C.

A small sample (1.2 L) of Large C was diluted with 450 mL of inhibited water (0.01 M NaOH) to ~7 M sodium concentration to prepare material for a test run of Sr/TRU precipitation and ion exchange processes. The sodium, Sr^{90}, and free hydroxide concentrations were measured on this sample during the dilution. Any other needed characterization on this sample was completed by the downstream processes.

The remaining ~10.1 L of Large C sample was diluted with ~ 6.3 L of inhibited water (0.01 M NaOH) to a sodium concentration of ~6 M resulting in 7 bottles of diluted material with a total volume of 16.4 L. The sodium concentration was measured for each of the bottles of diluted Large C. Any other needed characterization on this sample was completed by the downstream processes. The diluted material was sent on to the Sr/TRU process for precipitation and filtration.

Sample Preparation

A 12 to 30-fold dilution with deionized, distilled water was generally necessary to lower the radiation levels on filtered supernatant samples before submittal for analysis. Standards were not submitted with the supernatant samples, however, the Analytical Development Section periodically measures standards and blanks to check the calibration and background of the instruments. The total dried solids for the sample were obtained by thoroughly mixing the sample and any insoluble solids present by vigorously shaking the bottle, removing an aliquot of the sample, and drying the aliquot at 100 °C to constant weight to remove free water. The dried insoluble solids were obtained by filtering a portion of the sample through a 0.45 μ filter and washing the solids obtained with a small amount of 0.01 M NaOH to remove interstitial supernatant liquid. The washed insoluble solids were then dried at 100 °C to constant weight to remove free water. Dissolution of samples of total dried solids and dried insoluble solids were performed in triplicate by contacting with aqua-regia or by fusion with sodium peroxide. The digested solids samples were diluted to 250 mL with deionized, distilled water before analysis. Quality control included dissolving a glass standard of known composition concurrently with the dried solids samples. The glass standard indicates potential problems with sample contamination or incomplete dissolution during the digestion procedure and systematic problems with the analytical procedures. Unless otherwise noted the glass standards showed a successful dissolution and accurate analyses.

Analytical Methods

Analytical Development Section (ADS) performed all analytical measurements with the exception of the weight percent solids and density measurements conducted in the Shielded Cells. ADS uses the following analytical methods for determination of specific species. Nitrate, nitrite, sulfate, oxalate, phosphate, formate, chloride, and fluoride were measured by ion chromatography (IC). Chloride and fluoride were also determined by the ion selective electrode (ISE) method. Aluminate, carbonate, and hydroxide were measured using a titration method employing $SrCl_2$ to precipitate carbonate allowing the determination of all three species. Sodium, aluminum, and iron, as well as other metallic elements, were measured using inductively-coupled plasma-emission spectroscopy (ICP-ES). Potassium and mercury were measured using atomic adsorption spectroscopy (AA) with mercury determined using the cold-vapor technique (CV). Gamma emitting fission products were measured using gamma spectroscopy. Actinides were determined by inductively-coupled plasma mass spectrometry (ICP-MS) and alpha counting spectroscopy. Sr^{90} was determined from beta liquid scintillation counting. Tc^{99} was measured by ICP-MS and ICP-ES.

Organic Analysis

Organic compounds were analyzed with three methods: Complexants such as EDTA and HEDTA were analyzed with ion-pair chromatography (IPC), low molecular weight organic acids such as citrate, glycolate, formate and acetate were analyzed with ion-exclusion chromatography (IEC), and IDA was analyzed with GC/MS. All the chromatographic conditions used at SRTC were identical to the ones developed by Jim Campbell and his coworkers at PNNL.[6] Sample preparations were modified as described below. Cesium removal was not conducted on the 241-AN-102 sample prior to analysis. Samples received for organic analysis had previously been diluted 30-fold with deionized water to allow removal from the Shielded Cell facility.

For analysis of HEDTA and EDTA by IPC, 4 mL of the sample was transferred by pipette into a 5-mL volumetric flask. Twelve drops of 2 N phosphoric acid was added to bring the pH of the sample to 6.5-7. Additionally, 50 µL of 0.5 M $CuSO_4$ solution was added to the sample. Deionized water was used to bring the volume to 5 mL. The resulting liquid was transferred to a vial and heated at 60 °C for 1 hour. This procedure produced consistent recovery's for HEDTA. The liquid was filtered and analyzed with the IPC method. Recovery efficiencies for HEDTA and EDTA were determined to be 0.49 and 0.66, respectively, using a 241-AN-107 simulant. Although the recoveries for HEDTA and EDTA were not 100% they were consistent over multiple runs over two different days. The accuracy of the recovery efficiencies was conservatively set at 10% based on the simulant tests. The recovery efficiencies were used to calculate the results for HEDTA and EDTA in the 241-AN-102 samples. The addition of a heating step to speed up the Cu(II) complexation and correction for recovery were the modifications of the original PNNL procedures.

Samples for analysis of organic acids by IEC were diluted 5-fold in a radio hood followed by filtration through a 0.2 µ syringe filter. The filtrate was analyzed with the IEC method.

Weight Percent Solids Analysis

The weight percent of total solids in the samples were measured using a conventional drying oven at 100 °C and stainless steel or polymethylpentene beakers. The weight percent of dissolved solids in a sample of the filtered supernate were measured in the same manner. The weight percent insoluble solids and soluble solids in the sample were calculated from the measurements of the weight percent total solids of the sample and the weight percent dissolved solids in the filtered supernate. Obtaining the weight percent solids analysis of samples in this manner avoids difficulties associated with reproducibly measuring the insoluble solids directly. For samples with less than 3 wt % insoluble solids a direct measurement is required. Equations 1 and 2 allow calculation of the weight percent of insoluble and soluble solids from the total solids and dissolved solids measurements. The weight percent of soluble solids gives the mass of the dissolved solids in the supernate expressed as a percentage of the mass of the sample. The weight percent of insoluble solids represents the mass of insoluble solids expressed as a percentage of the mass of the sample. A 15 wt % NaCl standard solution was measured concurrently

during the analysis of the samples. All measurements of the 15 wt % NaCl standard solution were within 5 % of the expected value.

w_{ds} = weight fraction of dissolved solids (wt dissolved solids/ wt of supernate)
w_{ts} = weight fraction of total solids (wt total solids/ wt of sludge slurry)
w_{is} = weight fraction of insoluble solids (wt insoluble solids/ wt of sludge slurry)
w_{ss} = weight fraction of soluble solids (wt dissolved solids/ wt of sludge slurry)

$$w_{is} = (w_{ts} - w_{ds}) / (1 - w_{ds}) \quad \text{Eq. 1}$$

$$w_{ss} = w_{ts} - w_{is} \quad \text{Eq. 2}$$

Density Measurements

Density measurements were made on both the total sample and the filtered supernate using a pipette tip with the small end heat-sealed. After heat sealing, these pipette tips provide a reproducible volume of 8.25 mL. The sample does not wet the pipette tips eliminating problems with entrained air bubbles when filling a narrow cylinder with thick slurries.

RESULTS

The Small C sample obtained from the first shipment of 241-AN-102 received at SRTC contained a concentrated salt solution with low insoluble solids as evidenced by the weight percent solids data in Table 3. The extremely low insoluble solids content in the as-received sample lead to difficulties in measuring the weight percent insoluble solids. Direct measurement of the weight percent insoluble solids on small sub-samples were inconclusive. The value in Table 3 of <0.1 wt% indicates the level of insoluble solids that should be detectable using a 50 mL sample.

After diluting the Small C sample with 0.01 M NaOH to a sodium concentration of ~6 M, another measurement of the weight percent solids was conducted. Table 3 also contains these results. The same problems encountered with determination of the weight percent insoluble solids on the as-received sample were encountered with the diluted sample. Due to the low insoluble solids content the dilution was achieved in a single step since minimal dissolution of the solids was observed. Table 4 shows the initial as-received and after dilution concentrations of sodium, Sr^{90} and free hydroxide in the Small C sample.

The analytical results for non-radioactive components of the filtered supernatant liquid after dilution, shown in Table 5, indicate a sodium level of 6.42 M in good agreement with the measurement made during the dilution. However, the free hydroxide concentration exceeds the value previously measured during the dilution by more than a factor of two. The value for the free hydroxide in Table 5 appears to be in error as another measurement made on a different sub-sample of Large C also indicated a lower hydroxide concentration (see Table 11). The aluminate and phosphate measurements

produced values 15-20% lower than the aluminum and phosphorus measurements made by the ICP-ES method. The carbonate value in Table 5 , the result of a single measurement, does not agree with the TIC result. The TIC result converted to a carbonate concentration exceeds the measured carbonate value by ~60%. The other two replicates were below detection limits for carbonate indicating a concentration of <0.3 M. The aluminate and carbonate measured by titration after treatment with $SrCl_2$ may be subject to potential error due to precipitation of other anions along with the carbonate or incomplete carbonate precipitation. The precipitation of aluminate and other anions along with the carbonate would lower the observed aluminate concentration and produce a high bias for the carbonate measurement assuming complete precipitation of carbonate. The method dilutes the sample by ~40X which should reduce the potential of significant precipitation of other anions. Precipitation of aluminate has not been observed with a standard containing.0.5 M aluminate, 0.5 M carbonate , and 0.5 M free hydroxide using the $SrCl_2$ method. The precipitation of aluminate may also reduce the free hydroxide concentration. In this sample, assuming a reliable TIC result, incomplete precipitation of the carbonate by the $SrCl_2$ may account for the low carbonate results. The oxalate and formate anions account for only 10% of the TOC in the sample. The remaining organic carbon results from the presence of organic complexants discussed below. The cation/anion balance appears reasonable (within 10%) using the higher 0.82 M free hydroxide value and the carbonate value listed in the table. However, using the lower free hydroxide value of 0.38 M determined during the dilution the difference between the cation and anion concentrations increases to 16%. Adding in the organic complexants discussed later in this report brings the cation/anion difference using the lower hydroxide to within 10%. Using both the lower free hydroxide value and lower TIC result as a carbonate concentration, the cation/anion difference rises to 26%. Adding in the organic complexants to the anion value only lowers the difference to ~20%. The cation/anion balance provides an indication of whether any significant species were missed or whether bias was present in the analytical methods for cations versus anions.

As shown in Table 6 the diluted Small C sample contains very high levels of soluble Sr^{90} due to the presence of the organic complexants. The value for Sr^{90} of 53.1 μCi/mL shows good agreement with the value of 58 μCi/mL in Table 4 measured at the completion of the dilution of the Small C sample. The Cm^{244} concentration measured in the sample appears to be elevated due to an inadvertent introduction of contamination into the 4 L bottle containing the first shipment of 241-AN-102 sample. As evidence for the contamination of the Small C sample, Table 10 shows the measurements of the Cm^{244} concentration in Large C after final compositing. The final compositing mixed the remaining 2.5 L of material in the first shipment with the other three shipments to provide a homogeneous Large C sample. The measured Cm^{244} levels in the undiluted Large C samples, as well as the plutonium isotopes and Am^{241}, were found to be two orders of magnitude lower than measured in the Small C sample. The concentrations of these species would be further lowered by dilution.

The total solids and insoluble solids of the diluted Small C sample of 241-AN-102 were fully characterized and the results shown in Tables 7 and 8. The total solids includes the insoluble solids present in the sample as well as all of the dissolved salts. The insoluble

solids were collected by filtration of the sample and washed with small portions of 0.01 M NaOH to remove interstitial supernatant liquid. For most of the elements listed in Tables 7 and 8 the values represent the average of six replicate samples, three from the aqua-regia dissolution and three from the sodium peroxide fusion method. In some cases, only aqua-regia data or the data from the sodium peroxide fusion method were used. For example, only the sodium peroxide fusion data was used to calculate an average silicon value because silicon does not dissolve well with the aqua-regia method. Sodium data cannot be obtained from the sodium peroxide fusion method so only the aqua-regia data was used to calculate an average value. A standard glass of known composition dissolved along with the sample provides an indication of the quality of the dissolution and the presence of contamination introduced during the dissolution procedure.

The data in Table 7 indicates that the sodium concentration at 27.8 wt % dominates the total solids analysis. Other metals of significant concentration in the total solids include aluminum, potassium, and phosphorus. In contrast, the insoluble solids contain high concentrations of aluminum, chromium, and iron in addition to sodium.

The radionuclide data in Table 8 was obtained through counting techniques and ICP-MS. Gamma spectroscopy identified only Cs^{137} in the total solids but also detected europium isotopes in the insoluble solids. The aqua-regia dissolution data for the total dried solids sample indicated uranium contamination of the sample during sample preparation as evidenced by the presence of significant concentrations of U^{235} and U^{238} in the glass standard. The poor precision of the aqua-regia data for uranium coupled with aqua-regia values an order of magnitude higher than the sodium peroxide fusion data provided further evidence of sample corruption with respect to uranium. Therefore, the aqua-regia data was not used to calculate average values for the uranium isotopes. U^{235} was not detected in the sodium peroxide fusion ICP-MS data. The plutonium isotopics between the total dried solids sample and the insoluble solids sample do not show good agreement indicating a problem with the data at least with respect to the two method used for obtaining the data. A Pu^{240} value was obtained by subtracting the Pu^{239} value from the ICP-MS from the $Pu^{239/240}$ value from alpha counting. A ratio for Pu^{239} to Pu^{240} of ~0.4 on a mass basis was found in the total solids sample while the insoluble solids show a ratio of ~4.0. As discussed previously, the levels of Cm^{244} detected in the both the total solids and insoluble solids data may be elevated due to an inadvertent introduction of contamination into the 4 L bottle containing the first shipment of 241-AN-102 sample.

Table 9 shows the results of the analysis for organic complexants present in the diluted Small C sample. Glycolate and formate showed the highest concentrations in the Small C sample. The formate value obtained exceeds the previous result (see Table 5) by ~20%. The total organic carbon content of the species in Table 9 plus the value for oxalate from Table 5 calculates to 11,900 mg/L, approximately 30% lower than the TOC result from Table 5.

The final compositing of the Large C sample mixed the remaining material in the first shipment in a single large bottle with the material from the last three shipments. After thoroughly mixing the material, the composite Large C sample was pumped into four

smaller bottles for ease of handling. Table 10 shows the results of weight percent solids measurements and select analyses on a single sample from each of the four composite bottles. The data shows relatively good consistency with the exception of the Pu^{238} and Cm^{244}. The Pu^{238} and Cm^{244} values measured for composite 1 appear to be significantly higher than the other three composite samples and accounts for the high percent relative standard deviation. Generally sodium measurements show a percent relative standard deviation of approximately 2%. The variability of the sodium data in Table 10 probably results from the large dilutions required to bring the sodium data to within the calibration range of the ICP-ES (<2000 mg/L) and matrix effects associated with high ionic strength solutions. Self absorption of the sodium signal in highly concentrated solution usually leads to a low bias on the result. The actinide data, obtained from counting methods, was found to be nearly two orders of magnitude lower than values obtained on the diluted Small C sample. The Large C values in Table 10 would be further lowered by a dilution to ~6 M sodium to match the dilution of the Small C sample. The Large C values for plutonium, americium, and curium isotopes provides evidence supporting the theory of contamination of the first shipment of 241-AN-102. The contaminated material in the first shipment diluted into a large volume of uncontaminated material would produce lower concentrations in the Large C composite.

Prior to diluting the bulk Large C sample, a small 1.2 L sample was diluted to ~7 M sodium to provide material for small scale tests of the Sr/TRU precipitation and ion exchange processes. Table 11 shows the sodium, Sr^{90}, and free hydroxide concentrations measured during the dilution of the sample. Comparing the values for Sr^{90} and free hydroxide in the final 7 M sodium solution with the final values for the dilution of the Small C sample indicates the Small C sample may have been slightly more concentrated than the final composite Large C sample.

After conducting the bulk dilution of the Large C sample to ~6 M sodium, the sodium concentration in each of the bottles was measured. The data in Table 12 indicates good consistency between the final sodium concentrations in each of the bottles. Back calculating the initial sodium concentration for each of the bottles based on the amount of inhibited water added and assuming no dissolution of solids results in an average sodium concentration of 10.1 M with a percent relative standard deviation of 2% for the seven bottles. The calculation provides evidence for the possible low bias of the sodium concentrations measured in the undiluted final composite in Table 10.

Table 13 shows a check on the self-consistency of the data for the Small C sample of 241-AN-102 diluted ~6 M sodium. The table was generated by converting the filtered supernatant data to a total dried solids concentration basis followed by adding the converted filtered supernatant and insoluble solids data together based on the weight percent solids and density data. Theoretically, the sum of these two samples should be equal to the total dried solids analysis minus any material leached from the insoluble solids during washing with 0.01 M NaOH. Analytical error, small errors in weight percent solids and density data, and the possibility that some material was washed from the insoluble solids combine to lead to large errors in the mass balance.

With the exception of sodium, potassium, aluminum and Cs^{137}, all predominantly soluble species present in high concentrations, the data indicates relatively poor agreement between the filtered supernatant liquid, the total dried solids, and the insoluble solids samples. The poorest agreement exists for species predominantly found in the insoluble solids at low concentrations. Most of the values with high percent differences show combined filtered supernatant and insoluble solids values lower then the total solids values indicating the possibility that some material was washed from the insoluble solids or a low weight percent insoluble solids value used in the calculation. The high Cm^{244} concentrations appear to be consistent through all three parts of the sample lending further evidence to the theory of contamination of the first shipment.

COMPARISON TO PREVIOUS ANALYSES OF 241-AN-102 SAMPLES

The 241-AN-102 samples received at SRTC were collected as grab samples in July and August of 1998 from the same riser location on the tank. Previous grab samples were obtained from tank 241-AN-102 initially in 1984 and again in 1988, 1989, 1995, and in February 1998. Two core samples were also taken in 1990. The documented results available for the same grab samples as those received at SRTC contains radionuclide and metal concentrations for the supernatant but no anion concentrations.[7] Analysis of the insoluble solids was also reported in the reference. Anion data for comparison to the current data was taken from the grab samples obtained in February 1998.[8] The supernatant results required adjustment because the reported data was for the as-received sample while the current effort analyzed a diluted supernatant. Average concentration values from the reference data were converted to molar concentrations and subsequently mathematically diluted using the ratio of the current data sodium concentration to the reference data sodium concentration (6.42/9.13). The calculation assumes no precipitation or dissolution of solids as a result of the dilution.

Table 14 shows the comparison between the data for the filtered supernatant liquid after dilution to ~6 M sodium, taken from Tables 5 and 6, and the calculated diluted composition of the data for the 1998 analysis of the same grab samples. The two sets of data show excellent agreement with the exception of a few low concentration species nearing the detection limits of the method. The good agreement results in part from the fact that the samples received at SRTC contained so few solids that dissolution of solids during dilution would have a negligible impact on the concentration of most species. The Cm^{244} and $Pu^{239/240}$ values from the current analysis, suspected of being high due to sample contamination, were found to be lower than the less-than values reported in the 1998 analysis as determined by ICP-MS. However, the alpha counting methods used to obtain results for Cm^{244} and $Pu^{239/240}$ have greater sensitivity than the ICP-MS method.

The comparison, in Table 15, of the anion data from the current analysis with results reported for the February 1998 grab samples shows relatively good agreement for most species. The TOC results, a very important measurement given the high organic content of the waste solution, show very good agreement. The average sodium content of the February 1998 grab samples as analyzed was 9.53 M. Therefore, a ratio of 6.42/9.53 was

used to mathematically dilute the composition to allow comparison. The much higher hydroxide concentration for the current analysis may result from a high bias due to incomplete carbonate precipitation by the $SrCl_2$/titration method and/or from the presence of the organic complexing agents.

Table 16 shows a comparison of the insoluble solids composition of the sample received at SRTC after dilution to ~ 6 M sodium with the solids composition reported in the 1998 analysis of the same grab samples. The data shows very poor agreement between the two solids compositions. With the exception of the usually highly soluble species, sodium, potassium, and Cs^{137}, the data from the current analysis shows higher concentrations in all cases. The large observed differences in the composition of the two samples results from the method for obtaining the solids from the sample. The solids from the 1998 analysis were isolated after centrifugation and decanting the supernatant liquid. The solids used in the current analysis were isolated by filtration after diluting the sample to ~6 M sodium. The solids were then washed on the filter with a small amount of 0.01 M NaOH to displace the interstitial supernatant liquid. The low sodium content of the current analysis indicates the presence of very little soluble salts in the dried solids. The high sodium content of the 1998 data indicates appreciable salt concentrations that would effectively dilute the concentration of the insoluble species.

CONCLUSIONS

The data obtained for the sample of 241-AN-102 received at SRTC appears relatively self-consistent for major components of the sample. However, the self-consistency of species at lower concentrations was relatively poor. Some inconsistency was observed between results were more than one method of determination was employed. The cation/anion balance from the analysis of the filtered supernatant liquid was found to be relatively poor also. Results for radionuclides present in low concentrations, namely the actinides, generally exhibit larger errors due to the difficulties in analyzing species present in low concentrations and the potential for introduction of small amounts of contamination during sample handling causing large uncertainties. Some evidence was found to indicate the contamination of the first shipment of 241-AN-102 samples received at SRTC with Cm^{244} and possibly Am^{241} and plutonium isotopes. An analysis of the organic complexants appears to be consistent with the TOC result. The comparison to previous analyses of samples from 241-AN-102 indicates general agreement with the current analytical results. The comparison of the solids analysis showed large deviations attributed to differences in obtaining the solids from the bulk sample.

Again, it should be stressed that the 241-AN-102 sample was made from combining several grab samples obtained from the same riser location out of a large non-homogenized waste tank. Large uncertainties exit as to how the results of the current analysis relate to the actual composition of the Hanford waste tank 241-AN-103 as a whole. The waste retrieval method employed could have a large impact on the composition of the waste feed stream from this tank since the tank contains solids which could dissolve during dilution.

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APPROVALS

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Table 1. Listing of Sample Labels from Bottles of 241-AN-102 Samples Received at SRTC.

Shipment No.	Jar No.	Jar Labels	
1st Shipment	Jar 1	2AN 98-43	S98T00-2183
	Jar 2	2AN 98-46	S98T00-2186
received	Jar 3	2AN 98-48	S98T00-2221
10/12/98	Jar 4	2AN 98-47	S98T00-2187
	Jar 5	2AN 98-49	S98T00-2214
	Jar 6	2AN 98-44	S98T00-2184
	Jar 7	2AN 98-50	S98T00-2189
	Jar 8	2AN 98-45	S98T00-2185
2nd Shipment	Jar 1	2AN 98-65	S98T00-2486
	Jar 2	2AN 98-61	S98T00-2480
received	Jar 3	2AN 98-59	S98T00-2479
11/9/98	Jar 4	2AN 98-60	S98T00-2482
	Jar 5	2AN 98-62	S98T00-2481
	Jar 6	2AN 98-64	S98T00-2485
	Jar 7	2AN 98-63	S98T00-2487
	Jar 8	2AN 98-66	S98T00-2484
3rd Shipment	Jar 1	2AN 98-67	S98T00-2483
	Jar 2	2AN 98-69	S98T00-2510
received	Jar 3	2AN 98-70	S98T00-2511
12/16/98	Jar 4	2AN 98-68	S98T00-2509
	Jar 5	2AN 98-LCOMP1	S98T00-2181A
	Jar 6	2AN 98-LCOMP3	S98T00-2181B
	Jar 7	2AN 98-72	S98T00-2513
	Jar 8	2AN 98-71	S98T00-2512
4th Shipment	Jar 1	2AN 98-58	S98T00-2478
	Jar 2	2AN 98-LCOMP8	S98T00-2181
received	Jar 3	2AN 98-LCOMP5	S98T00-2181
1/22/99	Jar 4	2AN 98-52	S98T00-2191
	Jar 5	2AN 98-LCOMP4	S98T00-2181
	Jar 6	2AN 98-53	S98T00-2188
	Jar 7	2AN 98-54	S98T00-2192
	Jar 8	2AN 98-51	S98T00-2190

Table 2. Descriptions of Source Material for the Parent Jar No.

Jar ID	Laboratory ID	Group Id	Sample Date	Type Sample Shipped
2AN-98-43	S98T002183	9800417	7/21/98	Unaltered Grab Sample
2AN-98-44	S98T002184	9800417	7/21/98	Unaltered Grab Sample
2AN-98-45	S98T002185	9800417	7/21/98	Unaltered Grab Sample
2AN-98-46	S98T002186	9800417	7/21/98	Unaltered Grab Sample
2AN-98-47	S98T002187	9800417	7/21/98	Unaltered Grab Sample
2AN-98-48	S98T002221	9800417	7/21/98	Unaltered Grab Sample
2AN-98-49	S98T002214	9800417	7/22/98	Unaltered Grab Sample
2AN-98-50	S98T002189	9800417	7/22/98	Unaltered Grab Sample
2AN-98-59	S98T002479	98000459	8/10/98	Unaltered Grab Sample
2AN-98-60	S98T002482	98000459	8/10/98	Unaltered Grab Sample
2AN-98-61	S98T002480	98000459	8/10/98	Unaltered Grab Sample
2AN-98-62	S98T002481	98000459	8/10/98	Unaltered Grab Sample
2AN-98-63	S98T002487	98000459	8/11/98	Unaltered Grab Sample
2AN-98-64	S98T002485	98000459	8/11/98	Unaltered Grab Sample
2AN-98-65	S98T002486	98000459	8/11/98	Unaltered Grab Sample
2AN-98-66	S98T002484	98000459	8/11/98	Unaltered Grab Sample
2AN-98-51	S98T002190	98000680	7/22/98	Unaltered Grab Sample
2AN-98-52	S98T002191	98000680	7/22/98	Unaltered Grab Sample
2AN-98-53	S98T002188	98000680	7/22/98	Unaltered Grab Sample
2AN-98-54	S98T002192	98000680	7/22/98	Unaltered Grab Sample
2AN-98-58	S98T002478	98000680	8/10/98	Unaltered Grab Sample
2AN-98-67	S98T002483	98000459	8/11/98	Unaltered Grab Sample
2AN-98-68	S98T002509	98000459	8/12/98	Unaltered Grab Sample
2AN-98-69	S98T002510	98000459	8/12/98	Unaltered Grab Sample
2AN-98-70	S98T002511	98000459	8/12/98	Unaltered Grab Sample
2AN-98-71	S98T002512	98000459	8/12/98	Unaltered Grab Sample
2AN-98-72	S98T002513	98000459	8/12/98	Unaltered Grab Sample
2AN-98-LCOMP1	S98T002181	98000087	7/21/98	Liquid Core Composite
2AN-98-LCOMP3	S98T002181	98000087	7/21/98	Liquid Core Composite
2AN-98-LCOMP4	S98T002181	98000087	7/21/98	Liquid Core Composite
2AN-98-LCOMP5	S98T002181	98000087	7/21/98	Liquid Core Composite
2AN-98-LCOMP8	S98T002181	98000087	7/21/98	Liquid Core Composite

Table 3. Results of Weight Percent Solids and Density Measurements for the first shipment Composite of 241-AN-102 Sample As-Received and for the Small C Sample Diluted to ~6 M Sodium.

	As Received 241-AN-102	241-AN-102 Diluted to 6.4 M Na
wt % Total Solids	50.3 (1)	37.3 (1)
wt % Dissolved Solids	50.3 (1)	37.7 (1)
wt % Insoluble Solids	<0.1*	<0.1*
wt % Soluble Solids	50.3**	37.3**
Density of Supernate, g/mL	1.47	1.33
Density of Slurry, g/mL	Not measured	1.34

Percent relative standard deviation for triplicate measurements are shown in parentheses.

*A direct measurement was made but the insoluble solids content was too low for an accurate measurement.
**Calculated from the difference between the wt % total solids and wt % insoluble solids.

Table 4. Behavior of Na, Sr, OH During Dilution of the Small C Sample of the 241-AN-102 Sample with 0.01 M NaOH to ~6 M Sodium.

	$[Na^+]$ M	Sr^{90} μCi/mL	$[OH^-]$ M	Total Volume
As Received	10.2	75	0.66	1.5 L
1st Dilution	6.37	58	0.38	2.25 L

Table 5. Concentration of Non-Radioactive Species in Filtered Supernatant of the Small C Sample of the 241-AN-102 Sample Diluted to ~6 M Sodium.

Element	Average Concentration mg/L	Average Concentration M	% Relative Standard Deviation
Ag	<3.04E-01	<2.82E-06	
Al	9.22E+03	3.42E-01	0.9
B	2.46E+01	2.28E-03	1.0
Ba	<3.06E-01	<2.23E-06	
Ca	3.10E+02	7.74E-03	0.5
Cd	3.77E+01	3.36E-04	0.7
Co	2.27E+00	3.85E-05	0.4
Cr	1.59E+02	3.06E-03	0.8
Cu	1.52E+01	2.39E-04	1.9
Fe	2.20E+01	3.94E-04	1.3
Hg	<1.45E-01	<7.22E-07	23
K	1.38E+03	3.52E-02	1.8
La	8.70E+00	6.27E-05	3.8
Li	<5.07E-01	<7.31E-05	
Mg	<1.20E+00	<4.95E-05	
Mn	1.10E+01	2.00E-04	0.6
Mo	3.65E+01	3.80E-04	0.8
Na	1.48E+05	6.42E+00	2.7
Ni	2.57E+02	4.38E-03	0.8
P	1.20E+03	3.87E-02	3.1
Pb	1.15E+02	5.56E-04	1.2
Si	7.45E+00	2.65E-04	8.0
Sn	1.30E+01	1.10E-04	3.4
Sr	1.79E+00	2.04E-05	0.5
Ti	<3.48E-01	<7.26E-06	
V	<2.32E-01	<4.55E-06	
Zn	3.48E+00	5.32E-05	7.0
Zr	1.07E+01	1.17E-04	22

Table 5. Concentration of Non-Radioactive Species in Filtered Supernatant of the Small C Sample of the 241-AN-102 Sample Diluted to ~6 M Sodium. (Continued)

Analyte	Average Concentration mg/L	Average Concentration M	% Relative Standard Deviation
$[NO_3^-]$	1.20E+05	1.94E+00	10
$[NO_2^-]$	5.20E+04	1.13E+00	10
$[PO_4^{3-}]$	3.14E+03	3.30E-02	10
$[SO_4^{2-}]$	8.15E+03	8.48E-02	15
$[C_2O_4^{2-}]$	3.73E+02	4.24E-03	1.5
$[Cl^-]$	2.40E+03	6.76E-02	4.4
$[F^-]$	1.38E+03	7.28E-02	5.8
$[CHO_2^-]$	6.79E+03	1.51E-01	11
$[OH^-]$ free	1.39E+04	8.17E-01	7.2
$[CO_3^{2-}]$	2.94E+04*	4.89E-01*	
$[AlO_2^-]$	1.62E+04	2.74E-01	27
TIC	9213.2	**	1.2
TOC	17049.7	**	8.2

*Result of a single measurement. The other two replicates were below detection limits indicating a concentration of <0.3 M.

**Cannot calculate molar concentrations without knowing the specific compounds represented by the analytical result.

Table 6. Concentration of Radioactive Species in Filtered Supernatant of the Small C Sample of the 241-AN-102 Sample Diluted to ~6 M Sodium.

Analyte	Average Concentration mg/L	Average Concentration µCi/mL	% Relative Standard Deviation
Sr^{90}	3.87E-01	5.31E+01	8.2
Tc^{99} *	6.28E+00	1.07E-01	1.5
Cs^{133} *	7.06E+00	stable	2.3
Cs^{135} *	2.08E+00	2.40E-03	3.1
Cs^{137} *	3.72E+00	3.23E+02	1.3
Cs^{137}	3.10E+00	2.70E+02	1.0
U^{235} *	1.14E-01	2.45E-07	15
U^{238} *	1.63E+01	5.47E-06	3.4
Np^{237} *	1.74E-01	1.22E-04	13
Pu^{239} *	6.58E-02	4.03E-03	11
Pu^{238}/Am^{241}	***	1.27E-01	18
$Pu^{239/240}$	***	1.63E-02	59
Cm^{244} *	3.47E-03**	2.80E-01**	12
Total Alpha	***	4.31E-01	12

*Values were determined by ICP-MS.
**The high levels of Cm^{244}, and possibly the plutonium isotopes and Am^{241}, appear to be elevated due to contamination of the sample.
***Cannot calculate mg/L concentrations without knowing the specific isotopes or ratio of isotopes represented by the analytical result.

Table 7. Concentration of Non-Radioactive Species in Total Dried Solids and Insoluble Dried Solids of the Small C Sample of the 241-AN-102 Sample after Dilution to ~6 M Sodium.

	241-AN-102 Total Solids		241-AN-102 Insoluble Solids	
Element	Average Concentration Wt%	% Relative Standard Deviation	Average Concentration Wt%	% Relative Standard Deviation
Al	1.73E+00	4.9	2.80E+01	3.2
B	<1.00E-02		<1.00E-02	
Ba	<8.00E-03		<3.00E-02	
Ca	1.31E-01	4.3	2.82E-01	73
Cd	6.62E-03	13	<2.00E-02	
Co	<1.50E-02		<1.40E-02	
Cr	3.97E-02	12	2.10E+00	1.6
Cu	<1.70E-02		<1.90E-02	
Fe	6.63E-02	16	1.40E+00	0.6
Hg	<1.00E-03		9.53E-04	2.7
K	2.27E-01	7.1	1.41E-02	0.4
La	<2.80E-02		<5.00E-02	
Li	<1.40E-02		<3.50E-02	
Mg	5.66E-03	35	<4.40E-02	
Mn	4.36E-03	45	<5.00E-01	
Mo	<8.00E-03		<1.00E-02	
Na	2.78E+01	2.4	1.95E+00	0.7
Ni	5.42E-02	10	<2.80E-02	
P	1.95E-01	36	1.25E-01	15
Pb	<6.30E-02		<2.00E-01	
Ru	<4.90E-02		<4.80E-02	
Si	<8.00E-02		2.21E-01	23
Sn	<1.70E-02		5.24E-02	56
Sr	<3.00E-03		<7.00E-03	
Tc	<1.80E-02		<2.60E-02	
Ti	<1.10E-02		<5.00E-02	
V	<1.60E-02		<1.50E-02	
Zn	9.48E-03	3.1	1.27E-01	2.2
Zr	<1.90E-02		<5.60E-02	

Table 8. Concentration of Radioactive Species in Total Dried Solids and Insoluble Dried Solids of the Small C Sample of the 241-AN-102 Sample after Dilution to ~6 M Sodium.

	241-AN-102 Total Solids		241-AN-102 Insoluble Solids	
Isotope	Average Concentration µCi/g	% Relative Standard Deviation	Average Concentration µCi/g	% Relative Standard Deviation
Sr^{90}	7.86E+01	11	1.16E+03	16
Tc^{99} *	2.30E-01	7.5	4.19E-02	22
Cs^{137}	4.48E+02	6.1	1.50E+01	9.2
Eu^{154}	Not detected		9.99E+00	21
Eu^{155}	Not detected		7.21E+00	23
U^{235} *	Not detected		5.05E-06	7.7
U^{238} *	8.90E-06	12	1.27E-04	4.5
Np^{237} *	Not detected		8.33E-03	9.1
Am^{241}	Not detected		6.83E+00	6.7
Pu^{238}/Am^{241}	1.38E+00	32	7.91E+00	18
$Pu^{239/240}$	8.96E-01	80	1.27E+00	68
Pu^{239} *	9.83E-02	67	6.53E-01	17
Pu^{240} *	Not detected		3.32E-01	15
Pu^{241} **	Not detected		1.17E-04	
Cm^{244} *	8.68E-01	44	1.54E+01	14

*Values determined by ICP-MS.
**Calculated from mass 241 in ICP-MS minus the mass of Am^{241} obtained from gamma counting

Table 9. Organic Analysis of the Filtered Supernatant of the Small C Sample of the 241-AN-102 Sample Diluted to ~6 M Sodium.

Organic Ion	Average Concentration mg/L	% Relative Standard Deviation
citrate	3765	3.0
glycolate	8220	3.3
formate	8179	5.4
acetate	627	6.5
HEDTA	4484	1.6
EDTA	5820	0.6
IDA	2450	2.7

Total carbon content of these species plus oxalate from IC is ~11900 mg/L which is 30% low versus TOC result in Table 5.

Table 10. Results of Weight Percent Solids and Select Analyses for the Large C Composite of 241-AN-102 Sample Before Dilution.

Analysis	Composite 1	Composite 2	Composite 3	Composite 4	Average
Wt% Total Solids	50.2	50.9	50.5	50.5	50.5 (0.6)
Wt% Dissolved Solids	50.0	51.3	50.5	50.3	50.5 (1.1)
$[Na^+]$ M	1.01E+01	8.72E+00	9.63E+00	9.51E+00	9.48E+00 (5.9)
Pu^{238} μCi/mL	2.45E-04	1.35E-04	1.31E-04	1.52E-04	1.66E-04 (32)
$Pu^{239/240}$ μCi/mL	2.25E-04	2.49E-04	2.21E-04	2.50E-04	2.36E-04 (6.5)
Am^{241} μCi/mL	7.39E-03	6.44E-03	6.76E-03	7.03E-03	6.90E-03 (5.8)
Cm^{244} μCi/mL	1.75E-02	3.12E-03	2.49E-03	2.84E-03	6.48E-03 (113)

Percent relative standard deviation for the four measurements are shown in parentheses.

Table 11. Behavior of Na, Sr, OH During Dilution of a 1.2 L Sample of Large C Composite Diluted to ~7 M Sodium with 0.01 M NaOH.

	$[Na^+]$ M	Sr^{90} μCi/mL	$[OH^-]$ M	Total Volume
As Received	9.48			1.2 L
1st Dilution	8.13	58	0.39	1.5 L
2nd Dilution	7.13	45	0.29	1.65 L

Table 12. Final Sodium Concentration for the 7 Bottles of Large C Composite Sample Diluted to ~6 M Sodium with 0.01 M NaOH.

Bottle No.	$[Na^+]$ M
1	5.75
2	6.06
3	6.03
4	6.37
5	6.31
6	6.02
7	6.04

Table 13. Comparison of the Concentrations in the Total Dried Solids Sample Versus the Sum of the Filtered Supernatant and Insoluble Dried Solids Samples of the Small C Sample of 241-AN-102 Diluted to ~6 M Sodium.

	Units	Total Dried Solids	Filtrate and Insoluble Solids	Percent Difference
Counting				
Cs^{137}	uCi/g	4.48E+02	5.43E+02	21%
Sr^{90}	uCi/g	7.86E+01	1.10E+02	40%
Alpha	uCi/g	2.55E+00	9.30E-01	64%
$Pu^{239/240}$	uCi/g	8.96E-01	3.60E-02	96%
Pu^{238}/Am^{241}	uCi/g	1.38E+00	2.76E-01	80%
Cm^{244}	uCi/g	8.68E-01	6.03E-01	30%
ICP-ES				
Al	wt%	1.73E+00	1.93E+00	12%
Ca	wt%	1.31E-01	6.32E-02	52%
Cd	wt%	6.62E-03	7.65E-03	16%
Cr	wt%	3.97E-02	3.74E-02	6%
Fe	wt%	6.63E-02	8.02E-03	88%
K	wt%	2.27E-01	2.77E-01	22%
Na	wt%	2.78E+01	2.97E+01	7%
P	wt%	1.95E-01	2.42E-01	24%
Zn	wt%	9.48E-03	1.03E-03	89%
ICP-MS				
U^{238}	wt%	2.65E-03	3.38E-03	27%
Pu^{239}	wt%	1.60E-04	1.60E-05	90%
Tc^{99}	wt%	1.35E-03	1.26E-03	6%

Table 14. Comparison of the Current Results for the Filtered Supernatant Liquid to the Mathematically Diluted Composition from the 1998 Analysis of the Same Grab samples.

Analyte	Units	Calculated Diluted Supernatant Liquid 1998 Analysis[6]	Filtered Diluted Supernatant from Tables 5 and 6	Percent Difference
Cs^{137}	μCi/mL	3.07E+02	2.70E+02	14%
Sr^{90}	μCi/mL	6.24E+01	5.31E+01	18%
U^{238}	μCi/mL	6.96E-06	5.47E-06	27%
Al	M	3.80E-01	3.42E-01	11%
B	M	2.55E-03	2.28E-03	12%
Ca	M	8.37E-03	7.74E-03	8%
Cd	M	3.83E-04	3.36E-04	14%
Cr	M	3.78E-03	3.06E-03	24%
Cu	M	2.55E-04	2.39E-04	7%
Fe	M	5.51E-04	3.94E-04	40%
K	M	3.97E-02	3.52E-02	13%
Mn	M	6.65E-04	2.00E-04	233%
Mo	M	3.75E-04	3.80E-04	1%
Na	M	6.42E+00	6.42E+00	0%
Ni	M	4.87E-03	4.38E-03	11%
P	M	3.92E-02	3.87E-02	1%
Pb	M	6.15E-04	5.56E-04	11%
Zr	M	7.17E-05	1.17E-04	39%
TOC	mg/L	2.11E+04	1.70E+04	24%

Table 15. Comparison of the Current Results for the Filtered Supernatant Liquid to the Mathematically Diluted Composition from the Analysis of the February 1998 Grab samples.

Analyte	Units	Calculated Diluted Supernatant Liquid Feb. 1998 Analysis[7]	Filtered Supernatant from Tables 5 and 6	Percent Difference
$[NO_3^-]$	M	2.53E+00	1.94E+00	31%
$[NO_2^-]$	M	1.36E+00	1.13E+00	20%
$[PO_4^{3-}]$	M	4.31E-02	3.30E-02	31%
$[SO_4^{2-}]$	M	1.09E-01	8.48E-02	28%
$[C_2O_4^{2-}]$	M	8.42E-03	4.24E-03	99%
$[Cl^-]$	M	7.95E-02	6.76E-02	18%
$[F^-]$	M	6.52E-02	7.28E-02	10%
$[OH^-]_{free}$	M	1.04E-01	8.17E-01	87%
TIC	mg/L	9.44E+03	9.21E+03	2%
TOC	mg/L	1.62E+04	1.70E+04	5%
Na	M	6.42E+00	6.42E+00	0%

Table 16. Comparison of the Current Results for the Dried Insoluble Solids to the Solids Composition from the 1998 Analysis of the Same Grab samples.

Analyte	Units	Solids Data from 1998 Analysis[6]	Insoluble Solids from Tables 7 and 8	Percent Difference
Cs^{137}	μCi/g	2.27E+02	1.50E+01	1413%
Sr^{90}	μCi/g	8.98E+01	1.16E+03	92%
U^{235}	μCi/g	2.46E-06	5.05E-06	51%
U^{238}	μCi/g	6.08E-05	1.27E-04	52%
Al	wt%	9.96E-01	2.80E+01	96%
Ca	wt%	3.45E-02	2.82E-01	88%
Cr	wt%	3.35E-02	2.10E+00	98%
Fe	wt%	1.76E-02	1.40E+00	99%
K	wt%	1.45E-01	1.41E-02	928%
Na	wt%	1.42E+01	1.95E+00	628%
P	wt%	1.06E-01	1.25E-01	15%
Si	wt%	5.17E-03	2.21E-01	98%
Zn	wt%	5.18E-04	1.27E-01	100%